30° eine Überlappung der p_z-Orbitale erschwert ist, doch deutlich verkürzt. Der hier gefundene Bor-Phenyl-Abstand stimmt auch recht gut mit dem der p-Bromphenylborsäure (1,54 Å) (Zvonkova & Gluškova, 1958) sowie dem des Phenylbordichlorids (1,52 Å) (Coffin & Bauer, 1955) überein. Beide Verbindungen sind ebenfalls planar gebaut. Vom durchschnittlichen C–C-Abstand mit 1,388 Å sind nur geringe Abweichungen festzustellen.

Die Fig. 2 und 3 zeigen die Packung der Moleküle in der Zelle. Das ganze Molekül steht senkrecht zur Spiegelebene und ist weitgehend planar, lediglich die beiden Ringebenen des Benzodioxaborols schliessen einen Winkel von 177,5° ein. Die Molekülsymmetrie ist m (C_s); die Abweichung von der höheren Symmetrie mm (C_{2v}) ist jedoch nur gering. Die Packung der Moleküle erscheint sinnvoll, denn der senkrechte Abstand zwischen zwei übereinanderliegenden Molekülen entspricht mit 3,5 Å gerade der doppelten van der Waalschen Dicke von Aromaten. Der kürzeste Abstand eines Boratoms zu einer Benzolebene ist mit 3,36 Å zu gross, um eine Wechselwirkung zu erlauben.

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Redetermination of the $Al_2(WO_4)_3$ Structure

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Abstract. $Al_2(WO_4)_3$, orthorhombic, *Pbcn*, 4 formula units in the cell, $a=12\cdot574$ (5), $b=9\cdot045$ (4), $c=9\cdot121$ (4) Å, (20 °C), forms colourless, transparent crystals. Atomic parameters, except for the z coordinate of O(2) and some temperature factors, are not significantly different from those found by Craig & Stephenson [*Acta Cryst.* (1968). B**24**, 1250–1255] but are more precisely determined.

Introduction. The $Al_2(WO_4)_3$ crystals were prepared by J. Gaaf of this Laboratory by heating a mixture of WO_3 and Al_2O_3 (in a molar ratio of 3:1) for 70 h at 1100 °C.

Systematic absences, from precession photographs, were: $hk0$, $h+k=2n+1$; $h0l$, $l=2n+1$; and $0kl$, $k=2n+1$. A triangular prismatic crystal, dimensions $0\cdot1\times0\cdot1\times0\cdot5$ mm, was mounted with [010] coin-

Table 1. *Final atomic parameters for* $Al_2(WO_4)_3$

Figures in parentheses here and in succeeding tables are the estimated standard deviations in the least significant digits.

Present work	x	y	z	B	(1968) Craig & Stephenson
W(1)	0	0·4745 (1)	$\frac{1}{4}$	0·66 (4)*	W(1)
W(2)	0·3554 (6)	0·3958 (9)	0·1179 (9)	0·62 (4)*	W(2)
Al	0·3806 (4)	0·2497 (7)	0·4668 (7)	0·53 (10)	Al
O(1)	0·1400 (12)	0·0894 (17)	0·0911 (19)	1·6 (3)	O(2)
O(2)	0·0651 (12)	0·3649 (17)	0·1218 (18)	1·6 (3)	O(4)
O(3)	0·2558 (11)	0·3172 (17)	0·0073 (18)	1·4 (3)	O(1)
O(4)	0·4069 (12)	0·0882 (18)	0·3377 (19)	1·5 (3)	O(6)
O(5)	0·4794 (11)	0·3194 (17)	0·0682 (19)	1·4 (3)	O(3)
O(6)	0·3318 (11)	0·3607 (17)	0·3058 (17)	1·2 (3)	O(5)

Anisotropic thermal parameters for the tungsten atoms ($\times 10^5$). The form of the anisotropic thermal ellipsoid is given by $\exp\,[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
W(1)	177 (7)	220 (16)	−17 (14)	0	9 (9)	0
W(2)	128 (6)	265 (13)	35 (13)	0 (5)	−10 (6)	38 (9)

* Values of the Debye–Waller isotropic temperature factors from the final isotropic least-squares refinement.

cident with the φ axis, on a Nonius automatic diffractometer. Cell dimensions were calculated from the θ, $-\theta$ settings of 15 low-order reflexions ($0{\cdot}23$ Å$^{-1} \le \sin\theta/\lambda \le 0{\cdot}33$ Å^{-1}).

In the range $0 \le \sin\theta/\lambda \le 0{\cdot}59$ Å^{-1} 683 reflexions, out of a possible number of 928, were observed, with a θ–2θ scan, Zr-filtered Mo K radiation, a scintillation counter and pulse-height discrimination. A reflexion was considered to be observed when $\frac{1}{2}I \ge \sigma(I) = [C + B_1 + B_2 + (0{\cdot}08I)^2]^{1/2}$, in which C is the total integrated count, B_1 and B_2 are the backgrounds measured at each side of the scan for half the scan time, and $I = C - B_1 - B_2$ is the net count.

An experimental azimuth-dependent absorption correction was derived (Furnas, 1966) from azimuthal scans of the $0k0$ reflexions ($k = 2,4,\ldots,10$). This correction and the θ-dependent absorption correction for a spherical crystal with $\mu R = 1{\cdot}4$ (Weber, 1969), together with the normal Lorentz–polarization correction were applied to the data set. Structure amplitudes were obtained on a common arbitrary scale.

The full-matrix least-squares refinement [function minimized: $\sum\omega(|F_o| - |F_c|)^2$, where $\omega = 1/\sigma^2(F_o)$] started from the $Sc_2(WO_4)_3$ parameters (Abrahams & Bernstein, 1966) and, with isotropic temperature factors, resulted in a conventional R (based on F, observed reflexions only) of 0·078, $R_w = 0{\cdot}092 = [\sum\omega(|F_o| - |F_c|)^2/\sum\omega(F_o)^2]^{1/2}$. Inclusion of Zachariasen's (1968) extinction parameter reduced R to 0·062 and R_w to 0·079. Introduction of anisotropic temperature factors for the tungsten atoms yielded the final values: $R = 0{\cdot}047$, $R_w = 0{\cdot}057$; error in an observation of unit weight = 1·18. Weighting analyses based on ranges of $\sin\theta/\lambda$ and of F_o indicated the weighting scheme to be correct. Allowance for anisotropic motion of the other atoms gave no significant improvement. Throughout the refinement neutral atomic scattering factors were used; those for Al were taken from *International Tables for X-ray Crystallography* (1962), those for O and W from the tables of Cromer & Waber (1965) and the anomalous dispersion terms of W were taken from those of Cromer (1965).

The final parameters are listed in Table 1. The atomic numbering scheme used is that of Abrahams & Bernstein. For comparison the scheme of Craig & Stephenson (C & S) is also given. Since the latter workers used a different origin, the operation $x, \frac{1}{2} - y, z$ should be applied to their final coordinates.*

* The table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30429 (4 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Selected interatomic distances* (Å) *in* $Al_2(WO_4)_3$

The results of the present work and those of Craig & Stephenson are listed in columns 1 and 2 respectively.

	1	2		1	2
W(1) tetrahedron			Al octahedron		
W(1)–O(2)	1·74 (2)*	1·77 (3)	Al—O(1)	1·86 (2)	1·74 (5)
W(1)–O(4)	1·75 (2)	1·76 (3)	Al—O(2)	1·88 (2)	1·87 (3)
O(2)–O(2′)	2·86 (3)	2·93 (6)	Al—O(3)	1·86 (2)	1·83 (4)
O(2)–O(4)	2·86 (2)	2·83 (4)	Al—O(4)	1·91 (2)	1·86 (4)
O(2)–O(4′)	2·84 (2)	2·92 (5)	Al—O(5)	1·90 (2)	1·89 (3)
O(4)–O(4′)	2·84 (3)	2·88 (7)	Al—O(6)	1·88 (2)	1·82 (4)
			O(1)–O(2)	2·68 (2)	2·75 (5)
W(2) tetrahedron			O(1)–O(3)	2·64 (2)	2·58 (5)
W(2)–O(1)	1·77 (2)	1·80 (4)	O(1)–O(5)	2·62 (2)	2·49 (5)
W(2)–O(3)	1·76 (2)	1·82 (3)	O(1)–O(6)	2·67 (2)	2·34 (8)
W(2)–O(5)	1·77 (2)	1·77 (3)	O(2)–O(3)	2·65 (2)	2·65 (4)
W(2)–O(6)	1·77 (2)	1·80 (4)	O(2)–O(4)	2·65 (2)	2·53 (5)
O(1)–O(3)	2·89 (2)	2·83 (5)	O(2)–O(5)	2·64 (2)	2·58 (5)
O(1)–O(5)	2·88 (2)	2·85 (5)	O(3)–O(4)	2·71 (2)	2·63 (5)
O(1)–O(6)	2·87 (2)	3·08 (6)	O(3)–O(6)	2·68 (2)	2·61 (5)
O(3)–O(5)	2·87 (2)	2·89 (4)	O(4)–O(5)	2·68 (2)	2·66 (4)
O(3)–O(6)	2·91 (2)	3·05 (5)	O(4)–O(6)	2·66 (2)	2·68 (4)
O(5)–O(6)	2·88 (2)	2·91 (5)	O(5)–O(6)	2·67 (2)	2·64 (4)

Metal–metal distances

	1	2
Al–W(1)	3·518 (8)	3·504 (16)
Al–W(1′)	3·614 (8)	3·635 (17)
Al–W(2)	3·464 (9)	3·442 (20)
Al–W(2′)	3·506 (9)	3·513 (18)
Al–W(2″)	3·529 (7)	3·543 (17)
Al–W(2‴)	3·657 (8)	3·650†

* Estimated standard deviations calculated from the variance–covariance matrix by *ORFFE* (Busing, Martin & Levy, 1964).

† Not given by Craig & Stephenson.

Table 3. *Selected bond angles in* $Al_2(WO_4)_3$

(Presentation of the results as in Table 2)

	1	2		1	2
W(1) tetrahedron			Al octahedron		
O(2)–W(1)–O(2′)	110·5 (10)	111·5*	O(1)–Al–O(2)	91·3 (7)	99·0 (23)
O(2)–W(1)–O(4)	110·0 (7)	106·4 (16)	O(1)–Al–O(3)	90·3 (7)	92·4 (17)
O(2)–W(1)–O(4′)	109·1 (8)	111·5 (14)	O(1)–Al–O(4)	177·8 (7)	174·0*
O(4)–W(1)–O(4′)	108·1 (10)	109·7 (19)	O(1)–Al–O(5)	88·4 (7)	86·3 (16)
			O(1)–Al–O(6)	90·8 (8)	82·0 (22)
W(2) tetrahedron			O(2)–Al–O(3)	90·3 (7)	91·7 (14)
O(1)–W(2)–O(3)	110·1 (7)	102·7 (18)	O(2)–Al–O(4)	88·8 (8)	85·4 (15)
O(1)–W(2)–O(5)	108·8 (5)	106·0 (16)	O(2)–Al–O(5)	88·5 (7)	86·6 (14)
O(1)–W(2)–O(6)	108·5 (7)	117·1 (22)	O(2)–Al–O(6)	177·1 (8)	176·8*
O(3)–W(2)–O(5)	108·9 (7)	106·9 (14)	O(3)–Al–O(4)	91·9 (8)	91·1 (14)
O(3)–W(2)–O(6)	111·3 (7)	114·4 (13)	O(3)–Al–O(5)	178·2 (8)	177·7*
O(5)–W(2)–O(6)	109·1 (7)	108·9 (15)	O(3)–Al–O(6)	91·6 (7)	91·3 (14)
			O(4)–Al–O(5)	89·4 (7)	90·4 (14)
			O(4)–Al–O(6)	89·0 (8)	93·3 (17)
			O(5)–Al–O(6)	89·6 (7)	90·5 (14)

* Value not given by Craig & Stephenson.

Discussion. The structure determination was undertaken since the estimations of the Al coordination which could be derived from the $Sc_2(WO_4)_3$ structure (Abrahams & Bernstein, 1966) were not sufficiently precise. It was only after the refinement was started that we learned of the work of C & S based on Weissenberg data. Since diffractometer data are likely to yield more precise results than photographic data, it was decided to continue the calculations.

The cell dimensions calculated agree within experimental error with those obtained by C & S and those found by Trunov, Lutsenko & Kovba (1967). A comparison of the atomic parameters of the two structure determinations shows that the e.s.d.'s obtained in this work are about half those reported by C & S. Furthermore, all atomic coordinates do not differ by more than three times the e.s.d.'s obtained by C & S except for O(1) [this work: $z=0{\cdot}091$ (2), C & S: $z=0{\cdot}058$ (7)]. This difference could be due to some disorder in the crystal used by C & S, which would also be consistent with the high temperature factor of 2·7 (8) Å^2 found by them for this atom. The O(1) position determined in the present structure results in a much smaller spread in the comparable interatomic distances and angles (see Tables 2 and 3).

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Cadmium(II) Formate Dihydrate

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Abstract. $C_4H_{12}O_{12}Cd_2$, monoclinic, $P2_1/c$, $a=8{\cdot}982$ (4), $b=7{\cdot}391(6)$, $c=9{\cdot}760(3)$ Å, $\beta=97{\cdot}32(3)°$, $Z=2$, $D_x=2{\cdot}44$ g cm^{-3}. As previously reported [Osaki, Nakai & Watanabé (1964). *J. Phys. Soc. Japan*, **19**, 717–723] the structure is isomorphous with the formates of manganese, zinc and copper, and forms a three-dimensional polymer. There are two formate ligands, exhibiting *anti–anti* and *anti–syn* configurations, linking together cadmium atoms which are arranged in face-centred positions in the cell. The polymer is further strengthened by hydrogen bonds between coordinated water molecules and formate oxygen atoms. Cd–O distances vary between 2·243(5) and 2·326(5) Å.

Introduction. Cadmium(II) formate was prepared by dissolving finely divided $CdCO_3$ in a slight excess of aqueous formic acid, and crystallized from aqueous solution as colourless parallelepipeds. Accurate cell dimensions were obtained from a least-squares treatment of the 2θ values of 16 reflexions measured on a General Electric XRD 6 diffractometer. Systematic absences were $h0l$ with l odd, $0k0$ with k odd; space group $P2_1/c$. For data collection, a crystal of size approximately $0{\cdot}20\times0{\cdot}20\times0{\cdot}15$ mm was mounted with **b** coincident with the instrument φ axis and all reflexions with $2\theta\leq55°$ measured with Zr-filtered Mo $K\alpha$ radiation and the θ–2θ scan technique at a speed of 2° min^{-1} in 2θ. Of a total 1456 observations, 890 had $I>3\sigma$ where $\sigma^2(I)=S+B+(0{\cdot}06S)^2$ (S=scan count, B=background) and were used in the structural refinement. Initial coordinates for the refinement were those of the isomorphous manganous formate dihydrate (Osaki, Nakai & Watanabé, 1964), and after three cycles with isotropic thermal parameters R was 0·099. Refinement was continued with anisotropic thermal parameters, and after two cycles R was 0·065. At this stage a difference synthesis indicated sites for all the hydrogen

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